Formation of diphosphates. A NMR study on the mechanism and stereochemistry of diphosphate formation from chiral dioxaphosphorinanes

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Published in:
Journal of the American Chemical Society

DOI:
10.1021/ja992770k

Publication date:
2000

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# ja992770k.cif

# CIF-file generated for  s691a

#=============================================================================
data_global
#=============================================================================



#=============================================================================

# 1. SUBMISSION DETAILS

_publ_contact_author_name           # Crystallographic correspondence author
;   Dr. A.L. Spek
;
_publ_contact_author_address        # Address of author for correspondence
;
    Bijvoet Center for Biomolecular Research
    Department of Crystal and Structural Chemistry
    Utrecht University
    Padualaan 8
    3584 CH Utrecht
    The Netherlands
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_publ_contact_author_email           'spea@chem.uu.nl'
_publ_contact_author_fax             '+31 30 2533940'
_publ_contact_author_phone           '+31 30 2532538'

_publ_requested_journal              'J. Am. Chem. Soc.'

#=============================================================================

# 3. TITLE AND AUTHOR LIST

_publ_section_title
;
he Formation of Pyrophosphates; An NMR Study on the Mechanism
and Stereochemistry of Pyrophosphate Formation from Chiral
Dioxaphosphorinanes
;

loop_
_publ_author_name

'Ron Hulst'
'Johanna M. Visser'
'N. Koen de Vries'
'Robert W.J. Zijlstra'
'Auke Meetsma'
'Huub Kooijman'
'Wilberth J.J. Smeets'
'Anthony L. Spek'
'Ben L. Feringa'

#=============================================================================


#=============================================================================

data_s691a

#=============================================================================

# 5. CHEMICAL DATA

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;
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_chemical_formula_structural         ?
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_chemical_formula_weight                360.37
_chemical_compound_source            'see text'

loop_
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_atom_type_description
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_source
 P   P    0.1023    0.0942
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 O   O    0.0106    0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 H   H    0.0000    0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 C   C    0.0033    0.0016
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

#==============================================================================

# 6. CRYSTAL DATA

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_symmetry_space_group_name_Hall      'P 2ac 2ab'
_symmetry_space_group_name_H-M       'P 21 21 21'

loop_
_symmetry_equiv_pos_as_xyz
 x,y,z
 1/2-x,-y,1/2+z
 1/2+x,1/2-y,-z
 -x,1/2+y,1/2-z

_cell_length_a                         6.4799(10)
_cell_length_b                          17.165(2)
_cell_length_c                          17.189(2)
_cell_angle_alpha                              90
_cell_angle_beta                               90
_cell_angle_gamma                              90
_cell_volume                            1911.9(4)
_cell_formula_units_Z                   4
_cell_measurement_temperature          295
_cell_measurement_reflns_used        ?
_cell_measurement_theta_min          ?
_cell_measurement_theta_max          ?
_cell_special_details
; ?
;

_exptl_crystal_description           ?
_exptl_crystal_colour                colourless
_exptl_crystal_size_max             1.1
_exptl_crystal_size_mid             0.45
_exptl_crystal_size_min             0.18
_exptl_crystal_size_rad              ?
_exptl_crystal_density_meas          ?
_exptl_crystal_density_diffrn            1.252
_exptl_crystal_density_method       'Not Measured'
_exptl_crystal_F_000                       768
_exptl_absorpt_coefficient_mu             0.16
_exptl_crystal_density_meas_temp     ?
_exptl_absorpt_correction_type       ?
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# 7. EXPERIMENTAL DATA

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_diffrn_radiation_monochromator      ?

_diffrn_measurement_device_type      ?
_diffrn_measurement_method           ?
_diffrn_detector_area_resol_mean     ?

_diffrn_standards_number             ?
_diffrn_standards_interval_count     ?
_diffrn_standards_interval_time      ?
_diffrn_standards_decay_%            ?

loop_
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_diffrn_standard_refln_index_k
_diffrn_standard_refln_index_l
 ? ? ?

# number of measured reflections (redundant set)
_diffrn_reflns_number                 1966
_diffrn_reflns_av_R_equivalents       0.2271
_diffrn_reflns_av_sigmaI/netI         0.1039
_diffrn_reflns_limit_h_min               0
_diffrn_reflns_limit_h_max               8
_diffrn_reflns_limit_k_min             -22
_diffrn_reflns_limit_k_max               0
_diffrn_reflns_limit_l_min             -22
_diffrn_reflns_limit_l_max              22
_diffrn_reflns_theta_min              1.68
_diffrn_reflns_theta_max             25.00
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_diffrn_measured_fraction_theta_max  ?
_diffrn_measured_fraction_theta_full ?
_diffrn_reflns_reduction_process
;
 ?
;

# number of unique reflections
_reflns_number_total                    1958
# number of observed reflections (> n sig(I))
_reflns_number_gt                       1061
_reflns_threshold_expression         >2sigma(i)

_computing_data_collection           'Locally modified CAD4-Version 5 Software'
_computing_cell_refinement           'SET4 (de Boer & Duisenberg, 1984)'
_computing_data_reduction            'HELENA (Spek, 1997)'
_computing_structure_solution        ?
_computing_structure_refinement      'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics        ?
_computing_publication_material      'PLATON (Spek, 1990)'

#===============================================================================

# 8. REFINEMENT DATA

_refine_special_details
```

```
;
 Refinement on F^2 for ALL reflections except those
 flagged by the user for potential systematic errors.
 Weighted R-factors wR and all goodnesses of fit S
 are based on F^2, conventional R-factors R are based
 on F, with F set to zero for negative F^2. The
 observed criterion of F^2 > 2sigma(F^2) is used only
 for calculating -R-factor-obs etc. and is not
 relevant to the choice of reflections for refinement.
 R-factors based on F^2 are statistically about twice
 as large as those based on F, and R-factors based on
 ALL data will be even larger.
;
_refine_ls_structure_factor_coef     Fsqd
_refine_ls_matrix_type               full
_refine_ls_weighting_scheme          'calc w=1/[S'
_refine_ls_weighting_details
 ?
_refine_ls_hydrogen_treatment        'H-atom refinement: see text '
_refine_ls_extinction_method         none
_refine_ls_abs_structure_details
 'Flack H.D. (1983), Acta Cryst. A39, 876-881'
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_refine_ls_number_parameters               246
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_refine_ls_number_constraints        ?
_refine_ls_R_factor_all                 0.1531
_refine_ls_R_factor_gt                  0.0511
_refine_ls_wR_factor_ref                0.1001
_refine_ls_wR_factor_gt                 0.0867
_refine_ls_goodness_of_fit_ref           0.923
_refine_ls_restrained_S_all              0.923
_refine_ls_shift/su_max                  0.000
_refine_ls_shift/su_mean                 0.000
_refine_diff_density_max                 0.188
_refine_diff_density_min                -0.216
_refine_diff_density_rms                 0.048

#===============================================================================

# 9. ATOMIC COORDINATES AND THERMAL PARAMETERS

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_atom_site_type_symbol
_atom_site_thermal_displace_type
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_U_iso_or_equiv
 P1     P  Uani     0.8230(2)   0.11286(8)   0.48444(9)     1.000   0.0458(5)
 O1     O  Uani     0.7471(7)    0.1396(2)    0.4101(2)     1.000  0.0683(14)
 O2     O  Uani     0.7368(6)    0.1547(2)    0.5582(2)     1.000  0.0503(14)
 O3     O  Uani     1.0648(5)    0.1169(2)    0.4897(2)     1.000  0.0553(12)
 O4     O  Uani     0.7667(5)    0.0258(2)    0.5023(2)     1.000  0.0420(12)
 C1     C  Uani     1.1668(9)    0.0766(3)    0.5529(3)     1.000    0.057(2)
 C2     C  Uani     1.1047(8)   -0.0098(3)    0.5565(3)     1.000    0.044(2)
 C3     C  Uani     1.1708(9)   -0.0513(3)    0.4824(3)     1.000    0.061(2)
 C4     C  Uani     1.2102(8)   -0.0436(3)    0.6278(3)     1.000    0.067(2)
 C5     C  Uani     0.8677(8)   -0.0116(3)    0.5697(3)     1.000  0.0430(19)
 C6     C  Uani     0.7746(8)   -0.0918(3)    0.5778(3)     1.000  0.0420(19)
 C7     C  Uani     0.7020(8)   -0.1345(3)    0.5156(4)     1.000  0.0520(17)
 C8     C  Uani     0.6102(9)   -0.2061(3)    0.5272(4)     1.000    0.067(3)
 C9     C  Uani    0.5851(11)   -0.2346(4)    0.6000(5)     1.000    0.082(3)
 C10    C  Uani    0.6566(10)   -0.1935(4)    0.6634(4)     1.000    0.073(3)
 C11    C  Uani     0.7474(9)   -0.1207(3)    0.6526(3)     1.000    0.060(2)
 C12    C  Uani    0.8088(10)    0.2312(3)    0.5834(3)     1.000    0.056(2)
 C13    C  Uani    0.7085(10)    0.2459(3)    0.6624(3)     1.000    0.058(2)
```

```
 C14    C  Uani    0.7787(11)    0.3267(3)    0.6909(3)     1.000    0.081(3)
 C15    C  Uani    0.7516(11)    0.1802(3)    0.7176(3)     1.000    0.061(2)
 C16    C  Uani    0.9432(13)    0.1722(4)    0.7527(4)     1.000    0.078(3)
 C17    C  Uani    0.9846(14)    0.1087(5)    0.7992(4)     1.000    0.099(4)
 C18    C  Uani    0.8395(18)    0.0519(5)    0.8119(4)     1.000    0.101(4)
 C19    C  Uani    0.6483(14)    0.0601(5)    0.7784(4)     1.000    0.095(4)
 C20    C  Uani    0.6058(11)    0.1241(4)    0.7314(3)     1.000    0.071(3)
 H11    H  Uiso    1.1328(14)    0.1005(9)   0.5997(18)     1.000      0.0770
 H12    H  Uiso      1.309(5)    0.0803(3)    0.5465(3)     1.000      0.0770
 H31    H  Uiso      1.096(4)  -0.0278(14)   0.4365(13)     1.000      0.0910
 H32    H  Uiso      1.136(4)  -0.1084(16)    0.4865(8)     1.000      0.0910
 H33    H  Uiso      1.324(4)  -0.0452(15)   0.4752(10)     1.000      0.0910
 H41    H  Uiso      1.152(4)  -0.0181(16)   0.6765(14)     1.000      0.1010
 H42    H  Uiso      1.365(5)  -0.0334(17)   0.6246(10)     1.000      0.1010
 H43    H  Uiso      1.184(4)  -0.1021(17)   0.6300(11)     1.000      0.1010
 H51    H  Uiso    0.8376(16)   0.0165(13)     0.613(2)     1.000      0.0520
 H71    H  Uiso    0.7144(10)  -0.1156(10)     0.467(2)     1.000      0.0620
 H81    H  Uiso      0.565(3)  -0.2355(18)     0.484(3)     1.000      0.0800
 H91    H  Uiso      0.513(4)    -0.287(3)    0.6080(7)     1.000      0.0980
 H101   H  Uiso    0.6444(12)  -0.2141(12)     0.713(3)     1.000      0.0870
 H111   H  Uiso      0.792(2)  -0.0903(15)     0.697(2)     1.000      0.0720
 H121   H  Uiso    0.7648(16)   0.2738(14)   0.5437(14)     1.000      0.0750
 H122   H  Uiso      0.969(5)    0.2317(3)    0.5883(3)     1.000      0.0750
 H131   H  Uiso      0.543(8)    0.2483(3)    0.6536(5)     1.000      0.0700
 H141   H  Uiso      0.753(6)   0.3645(12)   0.6513(16)     1.000      0.1210
 H142   H  Uiso      0.923(5)    0.3255(7)     0.702(2)     1.000      0.1210
 H143   H  Uiso      0.704(5)   0.3404(12)   0.7368(19)     1.000      0.1210
 H161   H  Uiso      1.062(7)     0.217(2)    0.7434(6)     1.000      0.0930
 H171   H  Uiso     1.117(10)    0.1042(6)   0.8230(17)     1.000      0.1190
 H181   H  Uiso      0.870(3)     0.009(3)     0.843(2)     1.000      0.1210
 H191   H  Uiso      0.570(8)     0.031(3)    0.7852(8)     1.000      0.1130
 H201   H  Uiso      0.483(8)    0.1287(5)   0.7102(14)     1.000      0.0850

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_atom_site_aniso_U_33
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_atom_site_aniso_U_13
_atom_site_aniso_U_12
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  O1        0.094(3)   0.065(2)   0.046(2)    0.009(2)   -0.017(2)    0.009(3)
  O2        0.054(3)   0.039(2)   0.058(2)   -0.008(2)    0.000(2)    0.005(2)
  O3        0.050(2)   0.055(2)   0.061(2)    0.008(2)    0.005(2)   -0.003(2)
  O4        0.041(2)   0.039(2)   0.046(2)   -0.003(2)   -0.013(2)    0.004(2)
  C1        0.038(3)   0.067(4)   0.066(4)    0.002(3)   -0.004(3)   -0.007(3)
  C2        0.035(4)   0.052(4)   0.046(3)   -0.003(3)    0.000(3)    0.006(3)
  C3        0.050(4)   0.063(3)   0.069(4)   -0.002(3)    0.009(4)    0.010(3)
  C4        0.044(4)   0.080(4)   0.078(4)    0.018(3)   -0.021(4)    0.014(4)
  C5        0.044(4)   0.045(3)   0.040(3)    0.002(3)   -0.005(3)    0.005(3)
  C6        0.028(3)   0.039(3)   0.059(4)    0.010(3)    0.001(3)    0.012(3)
  C7        0.045(3)   0.047(3)   0.064(3)   -0.003(3)   -0.001(4)    0.004(3)
  C8        0.054(4)   0.047(4)   0.099(5)    0.003(4)   -0.003(4)    0.000(3)
  C9        0.071(5)   0.049(4)   0.125(6)    0.019(5)    0.017(5)   -0.003(4)
  C10       0.056(5)   0.071(5)   0.092(5)    0.037(4)    0.021(5)    0.013(4)
  C11       0.049(4)   0.067(4)   0.063(4)    0.011(4)    0.002(3)    0.002(4)
  C12       0.076(5)   0.040(3)   0.051(3)   -0.005(3)    0.003(4)    0.006(4)
  C13       0.060(4)   0.054(3)   0.061(4)   -0.013(3)   -0.004(4)    0.003(3)
  C14       0.108(6)   0.064(4)   0.070(4)   -0.026(3)    0.001(4)   -0.001(4)
  C15       0.070(5)   0.071(4)   0.042(3)   -0.016(3)    0.009(4)   -0.002(4)
  C16       0.083(6)   0.093(5)   0.057(4)    0.004(4)   -0.015(5)   -0.009(5)
  C17       0.099(7)   0.131(7)   0.067(5)    0.016(5)   -0.015(5)    0.007(7)
  C18       0.158(9)   0.103(6)   0.042(4)    0.009(4)    0.008(6)    0.003(7)
  C19       0.127(9)   0.085(6)   0.072(6)   -0.008(5)    0.035(6)   -0.036(6)
  C20       0.067(5)   0.078(5)   0.067(4)   -0.020(4)    0.009(4)   -0.011(5)

#===============================================================================

# 10. MOLECULAR GEOMETRY
```

```
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;
 Bond distances, angles etc. have been calculated using the
 rounded fractional coordinates. All esds are estimated
 from the variances of the (full) variance-covariance matrix.
 The cell esds are taken into account in the estimation of
 distances, angles and torsion angles
;

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_geom_bond_site_symmetry_1
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 P1      O1          1.444(4)                    .       .                   yes
 P1      O2          1.561(4)                    .       .                   yes
 P1      O3          1.571(4)                    .       .                   yes
 P1      O4          1.569(4)                    .       .                   yes
 O2      C12         1.459(6)                    .       .                   yes
 O3      C1          1.448(6)                    .       .                   yes
 O4      C5          1.477(6)                    .       .                   yes
 C1      C2          1.538(7)                    .       .                   no
 C2      C3          1.521(7)                    .       .                   no
 C2      C4          1.519(7)                    .       .                   no
 C2      C5          1.553(7)                    .       .                   no
 C5      C6          1.509(7)                    .       .                   no
 C6      C7          1.379(8)                    .       .                   no
 C6      C11         1.389(7)                    .       .                   no
 C7      C8          1.380(7)                    .       .                   no
 C8      C9         1.353(11)                    .       .                   no
 C9      C10        1.378(11)                    .       .                   no
 C10     C11         1.394(9)                    .       .                   no
 C12     C13         1.526(8)                    .       .                   no
 C13     C14         1.540(8)                    .       .                   no
 C13     C15         1.500(7)                    .       .                   no
 C15     C16        1.387(11)                    .       .                   no
 C15     C20         1.370(9)                    .       .                   no
 C16     C17        1.378(11)                    .       .                   no
 C17     C18        1.372(13)                    .       .                   no
 C18     C19        1.373(14)                    .       .                   no
 C19     C20        1.391(10)                    .       .                   no
 C1      H11           0.93(3)                   .       .                   no
 C1      H12           0.93(3)                   .       .                   no
 C3      H31           1.01(2)                   .       .                   no
 C3      H32           1.01(3)                   .       .                   no
 C3      H33           1.01(3)                   .       .                   no
 C4      H41           1.02(3)                   .       .                   no
 C4      H42           1.02(3)                   .       .                   no
 C4      H43           1.02(3)                   .       .                   no
 C5      H51           0.91(3)                   .       .                   no
 C7      H71           0.90(3)                   .       .                   no
 C8      H81           0.94(4)                   .       .                   no
 C9      H91           1.02(5)                   .       .                   no
 C10     H101          0.93(5)                   .       .                   no
 C11     H111          0.97(3)                   .       .                   no
 C12     H121          1.04(2)                   .       .                   no
 C12     H122          1.04(3)                   .       .                   no
 C13     H131          1.08(5)                   .       .                   no
 C14     H141          0.96(3)                   .       .                   no
 C14     H142          0.95(3)                   .       .                   no
 C14     H143          0.96(3)                   .       .                   no
 C16     H161          1.10(4)                   .       .                   no
 C17     H171          0.95(6)                   .       .                   no
 C18     H181          0.93(5)                   .       .                   no
 C19     H191          0.72(5)                   .       .                   no
 C20     H201          0.88(5)                   .       .                   no
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 O1      P1      O2         116.8(2)              .       .       .           yes
 O1      P1      O3         112.1(2)              .       .       .           yes
 O1      P1      O4         113.4(2)              .       .       .           yes
 O2      P1      O3         106.9(2)              .       .       .           yes
 O2      P1      O4         101.3(2)              .       .       .           yes
 O3      P1      O4       105.23(19)              .       .       .           yes
 P1      O2      C12        122.7(3)              .       .       .           yes
 P1      O3      C1         118.5(3)              .       .       .           yes
 P1      O4      C5         117.7(3)              .       .       .           yes
 O3      C1      C2         111.8(4)              .       .       .           yes
 C1      C2      C3         110.1(4)              .       .       .           no
 C1      C2      C4         106.4(4)              .       .       .           no
 C1      C2      C5         106.5(4)              .       .       .           no
 C3      C2      C4         111.7(4)              .       .       .           no
 C3      C2      C5         113.1(4)              .       .       .           no
 C4      C2      C5         108.6(4)              .       .       .           no
 O4      C5      C2         108.4(4)              .       .       .           yes
 O4      C5      C6         107.0(4)              .       .       .           yes
 C2      C5      C6         115.3(4)              .       .       .           no
 C5      C6      C7         123.3(5)              .       .       .           no
 C5      C6      C11        117.5(5)              .       .       .           no
 C7      C6      C11        119.0(5)              .       .       .           no
 C6      C7      C8         120.6(6)              .       .       .           no
 C7      C8      C9         120.5(6)              .       .       .           no
 C8      C9      C10        120.4(6)              .       .       .           no
 C9      C10     C11        119.7(6)              .       .       .           no
 C6      C11     C10        119.8(5)              .       .       .           no
 O2      C12     C13        106.1(4)              .       .       .           yes
 C12     C13     C14        107.8(5)              .       .       .           no
 C12     C13     C15        111.0(5)              .       .       .           no
 C14     C13     C15        114.9(5)              .       .       .           no
 C13     C15     C16        121.1(6)              .       .       .           no
 C13     C15     C20        120.6(6)              .       .       .           no
 C16     C15     C20        118.2(5)              .       .       .           no
 C15     C16     C17        120.3(7)              .       .       .           no
 C16     C17     C18        121.4(8)              .       .       .           no
 C17     C18     C19        118.6(8)              .       .       .           no
 C18     C19     C20        120.2(8)              .       .       .           no
 C15     C20     C19        121.3(7)              .       .       .           no
 O3      C1      H11       109.3(13)              .       .       .           no
 O3      C1      H12        109.3(6)              .       .       .           no
 C2      C1      H11       109.2(12)              .       .       .           no
 C2      C1      H12        109.3(6)              .       .       .           no
 H11     C1      H12        107.9(9)              .       .       .           no
 C2      C3      H31       109.4(14)              .       .       .           no
 C2      C3      H32       109.5(10)              .       .       .           no
 C2      C3      H33       109.4(12)              .       .       .           no
 H31     C3      H32       109.6(18)              .       .       .           no
 H31     C3      H33       109.6(19)              .       .       .           no
 H32     C3      H33          109(2)              .       .       .           no
 C2      C4      H41       109.4(15)              .       .       .           no
 C2      C4      H42       109.5(12)              .       .       .           no
 C2      C4      H43       109.4(13)              .       .       .           no
 H41     C4      H42       109.6(19)              .       .       .           no
 H41     C4      H43          109(2)              .       .       .           no
 H42     C4      H43          110(2)              .       .       .           no
 O4      C5      H51       108.5(16)              .       .       .           no
 C2      C5      H51        108.8(9)              .       .       .           no
 C6      C5      H51       108.8(15)              .       .       .           no
 C6      C7      H71       119.9(13)              .       .       .           no
 C8      C7      H71       119.6(14)              .       .       .           no
```

```
 C7      C8      H81          120(2)             .       .       .          no
 C9      C8      H81          120(2)             .       .       .          no
 C8      C9      H91        119.8(10)            .       .       .          no
 C10     C9      H91        119.8(11)            .       .       .          no
 C9      C10     H101       120.2(17)            .       .       .          no
 C11     C10     H101       120.1(17)            .       .       .          no
 C6      C11     H111       119.9(18)            .       .       .          no
 C10     C11     H111       120.3(18)            .       .       .          no
 O2      C12     H121       110.5(13)            .       .       .          no
 O2      C12     H122        110.5(5)            .       .       .          no
 C13     C12     H121       110.5(12)            .       .       .          no
 C13     C12     H122        110.6(6)            .       .       .          no
 H121    C12     H122        108.7(9)            .       .       .          no
 C12     C13     H131        107.7(7)            .       .       .          no
 C14     C13     H131        107.6(6)            .       .       .          no
 C15     C13     H131        107.5(7)            .       .       .          no
 C13     C14     H141       109.5(17)            .       .       .          no
 C13     C14     H142       109.5(10)            .       .       .          no
 C13     C14     H143       109.6(15)            .       .       .          no
 H141    C14     H142          109(3)            .       .       .          no
 H141    C14     H143          109(2)            .       .       .          no
 H142    C14     H143          110(3)            .       .       .          no
 C15     C16     H161       119.6(19)            .       .       .          no
 C17     C16     H161       120.1(19)            .       .       .          no
 C16     C17     H171       119.2(17)            .       .       .          no
 C18     C17     H171       119.4(16)            .       .       .          no
 C17     C18     H181       120.5(19)            .       .       .          no
 C19     C18     H181       120.9(19)            .       .       .          no
 C18     C19     H191          120(4)            .       .       .          no
 C20     C19     H191          120(4)            .       .       .          no
 C15     C20     H201       119.3(14)            .       .       .          no
 C19     C20     H201       119.4(15)            .       .       .          no

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_geom_torsion_atom_site_label_4
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_geom_torsion_site_symmetry_2
_geom_torsion_site_symmetry_3
_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
 O1      P1      O2      C12         -77.8(4)     .       .       .       .   no
 O3      P1      O2      C12          48.6(4)     .       .       .       .   no
 O4      P1      O2      C12         158.5(4)     .       .       .       .   no
 O1      P1      O3      C1         -166.5(3)     .       .       .       .   no
 O2      P1      O3      C1           64.3(4)     .       .       .       .   no
 O4      P1      O3      C1          -42.9(4)     .       .       .       .   no
 O1      P1      O4      C5          169.2(3)     .       .       .       .   no
 O2      P1      O4      C5          -64.8(4)     .       .       .       .   no
 O3      P1      O4      C5           46.4(4)     .       .       .       .   no
 P1      O2      C12     C13        -172.6(4)     .       .       .       .   no
 P1      O3      C1      C2           54.1(5)     .       .       .       .   no
 P1      O4      C5      C2          -59.7(5)     .       .       .       .   no
 P1      O4      C5      C6          175.4(3)     .       .       .       .   no
 O3      C1      C2      C5          -60.5(5)     .       .       .       .   no
 O3      C1      C2      C3           62.4(6)     .       .       .       .   no
 O3      C1      C2      C4         -176.3(4)     .       .       .       .   no
 C1      C2      C5      O4           62.2(5)     .       .       .       .   no
 C4      C2      C5      C6          -63.7(6)     .       .       .       .   no
 C1      C2      C5      C6         -178.0(4)     .       .       .       .   no
 C3      C2      C5      O4          -58.8(5)     .       .       .       .   no
 C3      C2      C5      C6           60.9(6)     .       .       .       .   no
 C4      C2      C5      O4          176.5(4)     .       .       .       .   no
 C2      C5      C6      C7          -90.0(6)     .       .       .       .   no
 C2      C5      C6      C11          95.1(6)     .       .       .       .   no
 O4      C5      C6      C11        -144.4(5)     .       .       .       .   no
 O4      C5      C6      C7           30.5(7)     .       .       .       .   no
```

```
 C11     C6      C7      C8          -2.2(8)     .       .       .       .   no
 C5      C6      C11     C10        178.1(5)     .       .       .       .   no
 C5      C6      C7      C8        -177.1(5)     .       .       .       .   no
 C7      C6      C11     C10          3.0(8)     .       .       .       .   no
 C6      C7      C8      C9           1.8(9)     .       .       .       .   no
 C7      C8      C9      C10         -2.0(10)    .       .       .       .   no
 C8      C9      C10     C11          2.7(10)    .       .       .       .   no
 C9      C10     C11     C6          -3.2(9)     .       .       .       .   no
 O2      C12     C13     C14       -179.4(4)     .       .       .       .   no
 O2      C12     C13     C15         53.9(6)     .       .       .       .   no
 C14     C13     C15     C16         -46.9(8)    .       .       .       .   no
 C14     C13     C15     C20        136.6(6)     .       .       .       .   no
 C12     C13     C15     C16         75.8(7)     .       .       .       .   no
 C12     C13     C15     C20       -100.7(7)     .       .       .       .   no
 C13     C15     C16     C17       -175.7(6)     .       .       .       .   no
 C13     C15     C20     C19        175.7(6)     .       .       .       .   no
 C16     C15     C20     C19         -0.9(9)     .       .       .       .   no
 C20     C15     C16     C17          0.9(10)    .       .       .       .   no
 C15     C16     C17     C18          0.2(11)    .       .       .       .   no
 C16     C17     C18     C19         -1.3(12)    .       .       .       .   no
 C17     C18     C19     C20          1.3(11)    .       .       .       .   no
 C18     C19     C20     C15         -0.2(10)    .       .       .       .   no

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_geom_contact_atom_site_label_1
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_geom_contact_distance
_geom_contact_site_symmetry_1
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 P1      C3         3.608(6)                     .       .                   no
 P1      H31         3.10(2)                     .       .                   no
 O2      C20        3.140(6)                     .       .                   no
 O3      C12        3.298(6)                     .     3_556                 no
 O1      H142        2.91(3)                     .     3_456                 no
 O1      H181        2.90(5)                     .     2_654                 no
 O1      H122        2.85(2)                     .     3_456                 no
 O2      H11       2.821(13)                     .       .                   no
 O2      H51         2.63(2)                     .       .                   no
 O3      H31         2.65(2)                     .       .                   no
 O3      H122      2.672(10)                     .       .                   no
 O3      H121        2.35(2)                     .     3_556                 no
 O3      H141        2.73(3)                     .     3_556                 no
 O4      H31         2.58(3)                     .       .                   no
 O4      H71       2.525(19)                     .       .                   no
 C1      C12        3.563(8)                     .       .                   no
 C3      P1         3.608(6)                     .       .                   no
 C3      C7         3.405(8)                     .       .                   no
 C4      C11        3.306(8)                     .       .                   no
 C7      C3         3.405(8)                     .       .                   no
 C11     C4         3.306(8)                     .       .                   no
 C12     O3         3.298(6)                     .     3_456                 no
 C12     C1         3.563(8)                     .       .                   no
 C20     O2         3.140(6)                     .       .                   no
 C1      H122      3.017(15)                     .       .                   no
 C4      H111        3.07(2)                     .       .                   no
 C6      H42         2.95(3)                     .     1_455                 no
 C6      H32         2.83(2)                     .       .                   no
 C6      H43         2.81(3)                     .       .                   no
 C7      H32         2.89(3)                     .       .                   no
 C7      H33         2.97(3)                     .     1_455                 no
 C9      H32         3.10(3)                     .     3_446                 no
 C10     H161        2.87(4)                     .     4_746                 no
 C10     H143        2.96(3)                     .     4_646                 no
 C11     H42         2.94(3)                     .     1_455                 no
 C11     H43         2.87(3)                     .       .                   no
 C12     H11       3.085(14)                     .       .                   no
 C14     H161        2.78(4)                     .       .                   no
 C16     H122         3.009(9)                   .       .                   no
 C16     H142      2.775(17)                     .       .                   no
```

C18	H71	2.90(3)	.	2_655	no
C19	H111	3.08(3)	.	.	no
H11	O2	2.821(13)	.	.	no
H11	C12	3.085(14)	.	.	no
H11	H41	2.43(3)	.	.	no
H11	H51	2.41(2)	.	.	no
H11	H122	2.50(2)	.	.	no
H12	H33	2.48(2)	.	.	no
H12	H42	2.40(3)	.	.	no
H31	P1	3.10(2)	.	.	no
H31	O3	2.65(2)	.	.	no
H31	O4	2.58(3)	.	.	no
H32	C6	2.83(2)	.	.	no
H32	C7	2.89(3)	.	.	no
H32	H43	2.49(2)	.	.	no
H32	C9	3.10(3)	.	3_546	no
H32	H91	2.55(4)	.	3_546	no
H33	C7	2.97(3)	.	1_655	no
H33	H12	2.48(2)	.	.	no
H33	H42	2.59(2)	.	.	no
H41	H11	2.43(3)	.	.	no
H41	H51	2.39(3)	.	.	no
H42	C6	2.95(3)	.	1_655	no
H42	C11	2.94(3)	.	1_655	no
H42	H12	2.40(3)	.	.	no
H42	H33	2.59(2)	.	.	no
H43	C6	2.81(3)	.	.	no
H43	C11	2.87(3)	.	.	no
H43	H32	2.49(2)	.	.	no
H43	H143	2.60(4)	.	4_746	no
H51	O2	2.63(2)	.	.	no
H51	H11	2.41(2)	.	.	no
H51	H41	2.39(3)	.	.	no
H51	H111	2.35(4)	.	.	no
H71	O4	2.525(19)	.	.	no
H71	C18	2.90(3)	.	2_654	no
H91	H32	2.55(4)	.	3_446	no
H101	H143	2.59(4)	.	4_646	no
H101	H161	2.36(5)	.	4_746	no
H111	C4	3.07(2)	.	.	no
H111	C19	3.08(3)	.	.	no
H111	H51	2.35(4)	.	.	no
H121	H141	2.42(3)	.	.	no
H121	O3	2.35(2)	.	3_456	no
H122	O3	2.672(10)	.	.	no
H122	C1	3.017(15)	.	.	no
H122	C16	3.009(9)	.	.	no
H122	H11	2.50(2)	.	.	no
H122	H142	2.55(3)	.	.	no
H122	O1	2.85(2)	.	3_556	no
H131	H201	2.305(19)	.	.	no
H141	H121	2.42(3)	.	.	no
H141	O3	2.73(3)	.	3_456	no
H142	C16	2.775(17)	.	.	no
H142	H122	2.55(3)	.	.	no
H142	H161	2.19(4)	.	.	no
H142	O1	2.91(3)	.	3_556	no
H143	C10	2.96(3)	.	4_656	no
H143	H43	2.60(4)	.	4_756	no
H143	H101	2.59(4)	.	4_656	no
H161	C14	2.78(4)	.	.	no
H161	H142	2.19(4)	.	.	no
H161	C10	2.87(4)	.	4_756	no
H161	H101	2.36(5)	.	4_756	no
H181	O1	2.90(5)	.	2_655	no
H201	H131	2.305(19)	.	.	no

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loop_
_geom_hbond_atom_site_label_D
_geom_hbond_atom_site_label_H
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_geom_hbond_atom_site_label_A
_geom_hbond_distance_DH
_geom_hbond_distance_HA
_geom_hbond_distance_DA
_geom_hbond_angle_DHA
_geom_hbond_site_symmetry_A
_geom_hbond_publ_flag
#
#D   H   A   D - H  H...A   D...A    D - H...A  symm(A)
#
C3      H31     O4         1.01(2)    2.58(3)   2.954(7)  101.3(16)     .   yes
C12     H121    O3         1.04(2)    2.35(2)   3.298(6)  150.7(16)   3_456 yes



# CIF-file generated for  S886B
#==============================================================================

data_S886B

#==============================================================================

# 5. CHEMICAL DATA

_chemical_name_systematic
;
 ?
;
_chemical_name_common                 ?
_chemical_melting_point               ?
_chemical_formula_moiety             'C19 H24 N O3 P'
_chemical_formula_structural          ?
_chemical_formula_sum                'C19 H24 N O3 P'
_chemical_formula_weight                 345.36
_chemical_compound_source            'see text'

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_atom_type_symbol
_atom_type_description
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_source
 P   P    0.2955    0.4335
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 O   O    0.0492    0.0322
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 N   N    0.0311    0.0180
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 H   H    0.0000    0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 C   C    0.0181    0.0091
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

#==============================================================================

# 6. CRYSTAL DATA

_symmetry_cell_setting                  Trigonal
_symmetry_space_group_name_Hall      'P 31'
_symmetry_space_group_name_H-M       'P 31'

loop_
_symmetry_equiv_pos_as_xyz
 x,y,z
 -y,x-y,1/3+z
 -x+y,-x,2/3+z

_cell_length_a                         12.4170(12)
_cell_length_b                         12.4170(12)
_cell_length_c                           10.912(2)
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_cell_angle_alpha                        90
_cell_angle_beta                         90
_cell_angle_gamma                       120
_cell_volume                        1457.0(3)
_cell_formula_units_Z                    3
_cell_measurement_temperature          295
_cell_measurement_reflns_used          ?
_cell_measurement_theta_min            ?
_cell_measurement_theta_max            ?
_cell_special_details
; ?
;

_exptl_crystal_description             ?
_exptl_crystal_colour                  colourless
_exptl_crystal_size_max               0.70
_exptl_crystal_size_mid               0.05
_exptl_crystal_size_min               0.05
_exptl_crystal_size_rad                ?
_exptl_crystal_density_meas            ?
_exptl_crystal_density_diffrn             1.181
_exptl_crystal_density_method         'Not Measured'
_exptl_crystal_F_000                       552
_exptl_absorpt_coefficient_mu             1.38
_exptl_crystal_density_meas_temp       ?
_exptl_absorpt_correction_type         ?
_exptl_absorpt_process_details         ?
_exptl_absorpt_correction_T_min       ?
_exptl_absorpt_correction_T_max       ?

#==============================================================================

# 7. EXPERIMENTAL DATA

_exptl_special_details
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_diffrn_radiation_wavelength             1.54184
_diffrn_radiation_type                 'Cu K\a'
_diffrn_radiation_source               ?
_diffrn_radiation_monochromator        ?

_diffrn_measurement_device_type        ?
_diffrn_measurement_method             ?
_diffrn_detector_area_resol_mean       ?

_diffrn_standards_number               ?
_diffrn_standards_interval_count       ?
_diffrn_standards_interval_time        ?
_diffrn_standards_decay_%              ?

loop_
_diffrn_standard_refln_index_h
_diffrn_standard_refln_index_k
_diffrn_standard_refln_index_l
 ? ? ?

# number of measured reflections (redundant set)
_diffrn_reflns_number                   5742
_diffrn_reflns_av_R_equivalents         0.0575
_diffrn_reflns_av_sigmaI/netI           0.0732
_diffrn_reflns_limit_h_min               -15
_diffrn_reflns_limit_h_max                15
_diffrn_reflns_limit_k_min               -15
_diffrn_reflns_limit_k_max                15
_diffrn_reflns_limit_l_min               -13
_diffrn_reflns_limit_l_max                13
_diffrn_reflns_theta_min                4.11
_diffrn_reflns_theta_max               74.85
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_diffrn_reflns_theta_full            ?
_diffrn_measured_fraction_theta_max  ?
_diffrn_measured_fraction_theta_full ?
_diffrn_reflns_reduction_process
;
 ?
;

# number of unique reflections
_reflns_number_total                   3828
# number of observed reflections (> n sig(I))
_reflns_number_gt                      2417
_reflns_threshold_expression         >2sigma(i)

_computing_data_collection           'Locally modified CAD4-Version 5 Software'
_computing_cell_refinement           'SET4 (de Boer & Duisenberg, 1984)'
_computing_data_reduction            'HELENA (Spek, 1997)'
_computing_structure_solution        ?
_computing_structure_refinement      'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics        ?
_computing_publication_material      'PLATON (Spek, 1990)'

#=============================================================================

# 8. REFINEMENT DATA

_refine_special_details
;
 Refinement on F^2 for ALL reflections except those
 flagged by the user for potential systematic errors.
 Weighted R-factors wR and all goodnesses of fit S
 are based on F^2, conventional R-factors R are based
 on F, with F set to zero for negative F^2. The
 observed criterion of F^2 > 2sigma(F^2) is used only
 for calculating -R-factor-obs etc. and is not
 relevant to the choice of reflections for refinement.
 R-factors based on F^2 are statistically about twice
 as large as those based on F, and R-factors based on
 ALL data will be even larger.
;
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_refine_ls_matrix_type               full
_refine_ls_weighting_scheme          'calc w=1/[S'
_refine_ls_weighting_details
 ?
_refine_ls_hydrogen_treatment        'H-atom refinement: see text '
_refine_ls_extinction_method         none
_refine_ls_abs_structure_details
 'Flack H.D. (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack              0.00(3)
_refine_ls_number_reflns                 3828
_refine_ls_number_parameters              223
_refine_ls_number_restraints                1
_refine_ls_number_constraints        ?
_refine_ls_R_factor_all                0.0907
_refine_ls_R_factor_gt                 0.0631
_refine_ls_wR_factor_ref               0.1678
_refine_ls_wR_factor_gt                0.1520
_refine_ls_goodness_of_fit_ref          0.983
_refine_ls_restrained_S_all             0.983
_refine_ls_shift/su_max                 0.008
_refine_ls_shift/su_mean                0.001
_refine_diff_density_max                0.189
_refine_diff_density_min               -0.181
_refine_diff_density_rms                0.045

#=============================================================================

# 9. ATOMIC COORDINATES AND THERMAL PARAMETERS
```

```
loop_
_atom_site_label
_atom_site_type_symbol
_atom_site_thermal_displace_type
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_U_iso_or_equiv
 P1     P  Uani   0.07639(10)  0.16672(10)   0.97802(9)     1.000   0.0641(4)
 O1     O  Uani     0.0045(3)    0.0326(3)    1.0050(3)     1.000  0.0856(12)
 O2     O  Uani     0.2038(3)    0.2367(3)    1.0492(2)     1.000  0.0764(12)
 O3     O  Uani     0.0108(2)    0.2436(3)    1.0184(2)     1.000  0.0628(11)
 N1     N  Uani     0.1094(4)    0.1962(4)    0.8361(4)     1.000  0.0746(14)
 C1     C  Uani    0.1215(13)    0.4702(8)   0.7112(12)     1.000    0.235(7)
 C2     C  Uani    0.0463(18)   0.5081(10)   0.6462(16)     1.000   0.295(10)
 C3     C  Uani   -0.0626(11)   0.4231(12)   0.6059(10)     1.000    0.159(5)
 C4     C  Uani    -0.0897(7)   0.3055(10)    0.6011(7)     1.000    0.137(4)
 C5     C  Uani    -0.0149(6)    0.2699(7)    0.6611(7)     1.000    0.110(3)
 C6     C  Uani     0.0918(6)    0.3548(5)    0.7181(4)     1.000  0.0792(18)
 C7     C  Uani     0.1780(5)    0.3186(4)    0.7813(4)     1.000  0.0758(16)
 C8     C  Uani     0.2752(6)    0.3210(7)    0.6922(6)     1.000    0.116(3)
 C9     C  Uani     0.1911(5)    0.2505(6)    1.1802(4)     1.000  0.0884(19)
 C10    C  Uani     0.1284(4)    0.3263(5)    1.2090(4)     1.000  0.0778(19)
 C11    C  Uani     0.2092(5)    0.4586(5)    1.1634(6)     1.000    0.100(2)
 C12    C  Uani     0.1148(6)    0.3239(8)    1.3481(5)     1.000    0.119(3)
 C13    C  Uani     0.0004(4)    0.2616(4)    1.1494(4)     1.000  0.0621(12)
 C14    C  Uani    -0.0708(4)    0.3297(4)    1.1598(4)     1.000  0.0657(16)
 C15    C  Uani    -0.1504(5)    0.3074(6)    1.2580(5)     1.000    0.091(2)
 C16    C  Uani    -0.2120(5)    0.3754(7)    1.2715(6)     1.000    0.106(3)
 C17    C  Uani    -0.1958(6)    0.4616(7)    1.1889(8)     1.000    0.112(3)
 C18    C  Uani    -0.1196(6)    0.4844(6)    1.0921(7)     1.000    0.107(3)
 C19    C  Uani    -0.0602(5)    0.4168(5)    1.0770(6)     1.000  0.0869(19)
 H1     H  Uiso    0.1936(13)    0.5300(8)   0.7497(12)     1.000       0.2820
 H1A    H  Uiso      0.079(5)     0.134(6)     0.777(6)     1.000       0.1120
 H2     H  Uiso    0.0744(18)   0.5920(10)   0.6326(16)     1.000       0.3520
 H3     H  Uiso   -0.1207(11)   0.4449(12)   0.5808(10)     1.000       0.1910
 H4     H  Uiso    -0.1588(7)   0.2473(10)    0.5575(7)     1.000       0.1640
 H5     H  Uiso    -0.0376(6)    0.1863(7)    0.6626(7)     1.000       0.1320
 H7     H  Uiso     0.2221(5)    0.3789(4)    0.8468(4)     1.000       0.0910
 H8A    H  Uiso      0.319(3)   0.4010(16)     0.653(3)     1.000       0.1740
 H8B    H  Uiso     0.2342(7)     0.258(3)     0.631(3)     1.000       0.1740
 H8C    H  Uiso      0.333(3)     0.306(5)    0.7366(9)     1.000       0.1740
 H9A    H  Uiso     0.2729(5)    0.2905(6)    1.2175(4)     1.000       0.1070
 H9B    H  Uiso     0.1428(5)    0.1686(6)    1.2166(4)     1.000       0.1070
 H11A   H  Uiso    0.2914(12)   0.4926(14)     1.197(3)     1.000       0.1500
 H11B   H  Uiso      0.174(2)   0.5083(11)     1.189(3)     1.000       0.1500
 H11C   H  Uiso      0.213(3)    0.4586(7)    1.0755(6)     1.000       0.1500
 H12A   H  Uiso     0.1957(6)     0.365(4)    1.3852(5)     1.000       0.1780
 H12B   H  Uiso      0.070(4)    0.2392(8)    1.3758(7)     1.000       0.1780
 H12C   H  Uiso      0.071(4)     0.366(4)    1.3707(6)     1.000       0.1780
 H13    H  Uiso    -0.0490(4)    0.1796(4)    1.1875(4)     1.000       0.0740
 H15    H  Uiso    -0.1630(5)    0.2468(6)    1.3154(5)     1.000       0.1100
 H16    H  Uiso    -0.2643(5)    0.3604(7)    1.3382(6)     1.000       0.1270
 H17    H  Uiso    -0.2372(6)    0.5062(7)    1.1980(8)     1.000       0.1340
 H18    H  Uiso    -0.1073(6)    0.5456(6)    1.0356(7)     1.000       0.1280
 H19    H  Uiso    -0.0110(5)    0.4311(5)    1.0079(6)     1.000       0.1040

loop_
_atom_site_aniso_label
_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
  P1       0.0712(7)  0.0632(7)  0.0603(5)   0.0034(5)   0.0142(5)   0.0353(6)
  O1        0.108(2)   0.061(2)   0.082(2)    0.012(2)    0.035(2)    0.038(2)
  O2        0.068(2)   0.100(2)   0.072(2)    0.000(2)  0.0054(15)    0.050(2)
  O3        0.061(2)   0.070(2) 0.0578(15) -0.0099(13)  0.0000(13)  0.0330(14)
```

```
 N1        0.089(3)   0.062(2)   0.065(2)    0.005(2)    0.022(2)    0.032(2)
 C1       0.343(15)   0.079(5)  0.285(13)   -0.057(6)  -0.195(12)    0.107(7)
 C2         0.41(2)   0.102(7)    0.40(2)  -0.069(10)    -0.24(2)   0.149(11)
 C3       0.202(10)   0.171(9)   0.175(8)    0.010(7)    0.018(7)    0.146(9)
 C4        0.109(5)   0.196(9)   0.116(5)    0.044(5)    0.022(4)    0.084(6)
 C5        0.108(4)   0.103(5)   0.117(4)    0.036(4)    0.010(4)    0.051(4)
 C6        0.116(4)   0.064(3)   0.059(2)   -0.004(2)    0.007(3)    0.046(3)
 C7        0.089(3)   0.064(3)   0.066(2)    0.009(2)    0.012(2)    0.032(2)
 C8        0.099(4)   0.134(5)   0.121(5)    0.051(4)    0.050(4)    0.062(4)
 C9        0.080(3)   0.131(4)   0.068(3)   -0.004(3)   -0.013(2)    0.063(3)
 C10       0.063(3)   0.104(4)   0.068(3)   -0.018(3)   -0.013(2)    0.043(3)
 C11       0.064(3)   0.093(4)   0.123(4)   -0.030(3)   -0.005(3)    0.025(3)
 C12       0.121(5)   0.185(7)   0.069(3)   -0.032(4)   -0.017(3)    0.091(5)
 C13       0.062(2)   0.064(2)   0.057(2)   -0.001(2)    0.011(2)    0.029(2)
 C14       0.057(2)   0.065(3)   0.069(3)   -0.013(2)   -0.001(2)    0.026(2)
 C15       0.077(3)   0.106(4)   0.092(4)   -0.013(3)    0.012(3)    0.047(3)
 C16       0.074(3)   0.145(6)   0.105(4)   -0.043(4)    0.006(3)    0.059(4)
 C17       0.083(4)   0.111(5)   0.153(6)   -0.039(5)   -0.018(4)    0.057(4)
 C18       0.088(4)   0.093(4)   0.151(6)    0.006(4)   -0.004(4)    0.053(3)
 C19       0.071(3)   0.086(3)   0.110(4)    0.000(3)    0.007(3)    0.044(3)

#===============================================================================

# 10. MOLECULAR GEOMETRY

_geom_special_details
;
 Bond distances, angles etc. have been calculated using the
 rounded fractional coordinates. All esds are estimated
 from the variances of the (full) variance-covariance matrix.
 The cell esds are taken into account in the estimation of
 distances, angles and torsion angles
;

loop_
_geom_bond_atom_site_label_1
_geom_bond_atom_site_label_2
_geom_bond_distance
_geom_bond_site_symmetry_1
_geom_bond_site_symmetry_2
_geom_bond_publ_flag
 P1      O1         1.473(3)                    .       .                   yes
 P1      O2         1.577(4)                    .       .                   yes
 P1      O3         1.596(3)                    .       .                   yes
 P1      N1         1.597(5)                    .       .                   yes
 O2      C9         1.458(5)                    .       .                   yes
 O3      C13        1.462(5)                    .       .                   yes
 N1      C7         1.449(6)                    .       .                   yes
 N1      H1A         0.93(7)                    .       .                   no
 C1      C2          1.43(3)                    .       .                   no
 C1      C6        1.291(11)                    .       .                   no
 C2      C3          1.31(2)                    .       .                   no
 C3      C4        1.325(17)                    .       .                   no
 C4      C5        1.377(13)                    .       .                   no
 C5      C6        1.363(10)                    .       .                   no
 C6      C7        1.518(10)                    .       .                   no
 C7      C8        1.538(10)                    .       .                   no
 C9      C10        1.524(9)                    .       .                   no
 C10     C12        1.526(7)                    .       .                   no
 C10     C13        1.522(8)                    .       .                   no
 C10     C11        1.518(8)                    .       .                   no
 C13     C14        1.502(7)                    .       .                   no
 C14     C19        1.364(8)                    .       .                   no
 C14     C15        1.389(8)                    .       .                   no
 C15     C16       1.402(10)                    .       .                   no
 C16     C17       1.335(11)                    .       .                   no
 C17     C18       1.350(12)                    .       .                   no
 C18     C19       1.377(10)                    .       .                   no
 C1      H1        0.930(18)                    .       .                   no
 C2      H2        0.930(16)                    .       .                   no
```

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 C3      H3          0.93(2)                    .       .                 no
 C4      H4        0.930(14)                    .       .                 no
 C5      H5        0.930(11)                    .       .                 no
 C7      H7         0.980(6)                    .       .                 no
 C8      H8A          0.96(2)                   .       .                 no
 C8      H8B          0.96(3)                   .       .                 no
 C8      H8C          0.96(4)                   .       .                 no
 C9      H9A        0.969(9)                    .       .                 no
 C9      H9B        0.970(9)                    .       .                 no
 C11     H11A         0.96(2)                   .       .                 no
 C11     H11B         0.96(2)                   .       .                 no
 C11     H11C       0.960(9)                    .       .                 no
 C12     H12A         0.96(3)                   .       .                 no
 C12     H12B       0.960(12)                   .       .                 no
 C12     H12C         0.96(5)                   .       .                 no
 C13     H13        0.980(6)                    .       .                 no
 C15     H15        0.930(9)                    .       .                 no
 C16     H16       0.930(10)                    .       .                 no
 C17     H17       0.930(12)                    .       .                 no
 C18     H18       0.930(10)                    .       .                 no
 C19     H19        0.930(9)                    .       .                 no

loop_
_geom_angle_atom_site_label_1
_geom_angle_atom_site_label_2
_geom_angle_atom_site_label_3
_geom_angle
_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_2
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
 O1      P1      O2          113.5(2)              .       .       .       yes
 O1      P1      O3          114.8(2)              .       .       .       yes
 O1      P1      N1          113.2(2)              .       .       .       yes
 O2      P1      O3        101.88(18)              .       .       .       yes
 O2      P1      N1          106.2(2)              .       .       .       yes
 O3      P1      N1          106.3(2)              .       .       .       yes
 P1      O2      C9          114.2(4)              .       .       .       yes
 P1      O3      C13         118.2(3)              .       .       .       yes
 P1      N1      C7          126.1(3)              .       .       .       yes
 C7      N1      H1A           112(4)              .       .       .       no
 P1      N1      H1A           122(4)              .       .       .       no
 C2      C1      C6         121.6(11)              .       .       .       no
 C1      C2      C3         119.0(11)              .       .       .       no
 C2      C3      C4         119.5(15)              .       .       .       no
 C3      C4      C5         119.6(10)              .       .       .       no
 C4      C5      C6          121.3(8)              .       .       .       no
 C1      C6      C7          120.2(8)              .       .       .       no
 C1      C6      C5          117.4(9)              .       .       .       no
 C5      C6      C7          122.3(5)              .       .       .       no
 N1      C7      C8          109.0(5)              .       .       .       yes
 C6      C7      C8          111.7(4)              .       .       .       no
 N1      C7      C6          111.5(5)              .       .       .       yes
 O2      C9      C10         113.2(4)              .       .       .       yes
 C9      C10     C12         106.2(5)              .       .       .       no
 C9      C10     C11         109.6(5)              .       .       .       no
 C11     C10     C13         112.4(4)              .       .       .       no
 C12     C10     C13         109.8(5)              .       .       .       no
 C9      C10     C13         107.9(4)              .       .       .       no
 C11     C10     C12         110.7(5)              .       .       .       no
 O3      C13     C10         110.5(4)              .       .       .       yes
 O3      C13     C14         106.4(4)              .       .       .       yes
 C10     C13     C14         115.1(4)              .       .       .       no
 C13     C14     C15         120.4(4)              .       .       .       no
 C15     C14     C19         116.5(5)              .       .       .       no
 C13     C14     C19         123.1(5)              .       .       .       no
 C14     C15     C16         120.6(6)              .       .       .       no
 C15     C16     C17         120.2(7)              .       .       .       no
 C16     C17     C18         120.3(8)              .       .       .       no
 C17     C18     C19         119.9(7)              .       .       .       no
```

```
 C14     C19     C18       122.4(6)            .       .       .          no
 C2      C1      H1       119.3(13)            .       .       .          no
 C6      C1      H1       119.1(16)            .       .       .          no
 C1      C2      H2          121(2)            .       .       .          no
 C3      C2      H2          121(2)            .       .       .          no
 C2      C3      H3       120.3(16)            .       .       .          no
 C4      C3      H3       120.2(15)            .       .       .          no
 C3      C4      H4       120.2(13)            .       .       .          no
 C5      C4      H4       120.1(12)            .       .       .          no
 C4      C5      H5       119.3(10)            .       .       .          no
 C6      C5      H5       119.4(10)            .       .       .          no
 N1      C7      H7        108.1(5)            .       .       .          no
 C6      C7      H7        108.2(6)            .       .       .          no
 C8      C7      H7        108.2(7)            .       .       .          no
 C7      C8      H8A         110(2)            .       .       .          no
 C7      C8      H8B      109.5(16)            .       .       .          no
 C7      C8      H8C      109.5(17)            .       .       .          no
 H8A     C8      H8B         109(3)            .       .       .          no
 H8A     C8      H8C         110(4)            .       .       .          no
 H8B     C8      H8C         110(3)            .       .       .          no
 O2      C9      H9A       109.0(6)            .       .       .          no
 O2      C9      H9B       108.9(6)            .       .       .          no
 C10     C9      H9A       109.0(6)            .       .       .          no
 C10     C9      H9B       108.9(7)            .       .       .          no
 H9A     C9      H9B       107.8(8)            .       .       .          no
 C10     C11     H11A     109.4(13)            .       .       .          no
 C10     C11     H11B     109.5(13)            .       .       .          no
 C10     C11     H11C      109.5(7)            .       .       .          no
 H11A    C11     H11B        109(2)            .       .       .          no
 H11A    C11     H11C        110(3)            .       .       .          no
 H11B    C11     H11C        109(3)            .       .       .          no
 C10     C12     H12A     109.5(10)            .       .       .          no
 C10     C12     H12B      109.4(8)            .       .       .          no
 C10     C12     H12C     109.5(11)            .       .       .          no
 H12A    C12     H12B        109(3)            .       .       .          no
 H12A    C12     H12C        109(3)            .       .       .          no
 H12B    C12     H12C        110(4)            .       .       .          no
 O3      C13     H13       108.2(4)            .       .       .          no
 C10     C13     H13       108.2(5)            .       .       .          no
 C14     C13     H13       108.3(6)            .       .       .          no
 C14     C15     H15       119.7(8)            .       .       .          no
 C16     C15     H15       119.6(8)            .       .       .          no
 C15     C16     H16       119.9(8)            .       .       .          no
 C17     C16     H16       119.9(9)            .       .       .          no
 C16     C17     H17      119.9(10)            .       .       .          no
 C18     C17     H17      119.8(10)            .       .       .          no
 C17     C18     H18       120.1(9)            .       .       .          no
 C19     C18     H18       120.0(9)            .       .       .          no
 C14     C19     H19       118.8(7)            .       .       .          no
 C18     C19     H19       118.8(7)            .       .       .          no

loop_
_geom_torsion_atom_site_label_1
_geom_torsion_atom_site_label_2
_geom_torsion_atom_site_label_3
_geom_torsion_atom_site_label_4
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_geom_torsion_site_symmetry_1
_geom_torsion_site_symmetry_2
_geom_torsion_site_symmetry_3
_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
 O1      P1      O2      C9        -71.4(4)      .       .       .       .   no
 O3      P1      O2      C9         52.5(4)      .       .       .       .   no
 N1      P1      O2      C9        163.6(4)      .       .       .       .   no
 O1      P1      O3      C13        70.3(3)      .       .       .       .   no
 O2      P1      O3      C13       -52.8(3)      .       .       .       .   no
 N1      P1      O3      C13      -163.8(3)      .       .       .       .   no
 O1      P1      N1      C7        179.5(5)      .       .       .       .   no
 O2      P1      N1      C7        -55.3(6)      .       .       .       .   no
```

```
 O3      P1      N1      C7        52.6(6)     .       .       .       .   no
 P1      O2      C9      C10      -61.4(5)     .       .       .       .   no
 P1      O3      C13     C14     -176.8(3)     .       .       .       .   no
 P1      O3      C13     C10       57.7(4)     .       .       .       .   no
 P1      N1      C7      C6      -101.8(5)     .       .       .       .   no
 P1      N1      C7      C8       134.4(5)     .       .       .       .   no
 C6      C1      C2      C3           9(2)     .       .       .       .   no
 C2      C1      C6      C5       -0.2(18)     .       .       .       .   no
 C2      C1      C6      C7      176.2(12)     .       .       .       .   no
 C1      C2      C3      C4         -15(2)     .       .       .       .   no
 C2      C3      C4      C5       13.3(17)     .       .       .       .   no
 C3      C4      C5      C6       -4.8(13)     .       .       .       .   no
 C4      C5      C6      C7      -178.0(7)     .       .       .       .   no
 C4      C5      C6      C1       -1.6(13)     .       .       .       .   no
 C1      C6      C7      N1       150.0(8)     .       .       .       .   no
 C5      C6      C7      C8        88.4(7)     .       .       .       .   no
 C5      C6      C7      N1       -33.8(7)     .       .       .       .   no
 C1      C6      C7      C8       -87.8(9)     .       .       .       .   no
 O2      C9      C10     C13       58.6(6)     .       .       .       .   no
 O2      C9      C10     C12      176.2(5)     .       .       .       .   no
 O2      C9      C10     C11      -64.1(6)     .       .       .       .   no
 C9      C10     C13     O3       -54.8(5)     .       .       .       .   no
 C9      C10     C13     C14     -175.2(4)     .       .       .       .   no
 C11     C10     C13     O3        66.2(5)     .       .       .       .   no
 C12     C10     C13     C14       69.5(6)     .       .       .       .   no
 C11     C10     C13     C14      -54.2(6)     .       .       .       .   no
 C12     C10     C13     O3      -170.1(5)     .       .       .       .   no
 O3      C13     C14     C15      146.5(5)     .       .       .       .   no
 C10     C13     C14     C19       87.9(6)     .       .       .       .   no
 O3      C13     C14     C19      -34.8(6)     .       .       .       .   no
 C10     C13     C14     C15      -90.9(6)     .       .       .       .   no
 C15     C14     C19     C18        3.3(9)     .       .       .       .   no
 C13     C14     C19     C18     -175.5(6)     .       .       .       .   no
 C13     C14     C15     C16      176.6(5)     .       .       .       .   no
 C19     C14     C15     C16       -2.2(9)     .       .       .       .   no
 C14     C15     C16     C17       0.8(10)     .       .       .       .   no
 C15     C16     C17     C18      -0.3(12)     .       .       .       .   no
 C16     C17     C18     C19       1.3(12)     .       .       .       .   no
 C17     C18     C19     C14      -2.9(11)     .       .       .       .   no

loop_
_geom_contact_atom_site_label_1
_geom_contact_atom_site_label_2
_geom_contact_distance
_geom_contact_site_symmetry_1
_geom_contact_site_symmetry_2
_geom_contact_publ_flag
 P1      H1A        3.05(8)                      .     2_555                no
 O1      N1        2.817(7)                      .     2_555                no
 O1      H9B       2.869(6)                      .       .                  no
 O1      H1A        1.90(8)                      .     2_555                no
 O1      H5       2.867(10)                      .     2_555                no
 O1      H9B       2.675(8)                      .     3_554                no
 O2      H7        2.765(5)                      .       .                  no
 O2      H11C     2.715(19)                      .       .                  no
 O3      H11C     2.668(18)                      .       .                  no
 O3      H19       2.477(7)                      .       .                  no
 N1      O1        2.817(5)                      .     3_554                no
 N1      H5       2.590(10)                      .       .                  no
 N1      H13       2.877(8)                      .     3_554                no
 C11     C19      3.258(10)                      .       .                  no
 C12     C15      3.343(11)                      .       .                  no
 C15     C12      3.343(11)                      .       .                  no
 C19     C11      3.258(10)                      .       .                  no
 C1      H8A        3.04(4)                      .       .                  no
 C1      H11A       3.10(2)                      .     3_564                no
 C4      H12C       3.06(3)                      .     1_554                no
 C5      H1A        2.79(7)                      .       .                  no
 C11     H1       3.081(16)                      .     2_665                no
 C11     H19      3.088(10)                      .       .                  no
```

C14	H12C	2.79(3)	.	.	no
C14	H11B	2.74(2)	.	.	no
C15	H12C	2.76(5)	.	.	no
C16	H3	2.994(16)	.	3_455	no
C17	H16	3.089(11)	.	3_454	no
C19	H11B	2.82(3)	.	.	no
H1	H7	2.331(14)	.	.	no
H1	C11	3.081(12)	.	3_564	no
H1	H11A	2.50(2)	.	3_564	no
H1A	C5	2.79(7)	.	.	no
H1A	H5	2.24(7)	.	.	no
H1A	H8B	2.38(7)	.	.	no
H1A	P1	3.05(7)	.	3_554	no
H1A	O1	1.90(7)	.	3_554	no
H2	H7	2.388(14)	.	3_564	no
H2	H11C	2.58(3)	.	3_564	no
H3	C16	2.994(19)	.	2_564	no
H5	N1	2.590(10)	.	.	no
H5	H1A	2.24(7)	.	.	no
H5	O1	2.867(9)	.	3_554	no
H7	O2	2.765(5)	.	.	no
H7	H1	2.331(14)	.	.	no
H7	H2	2.39(2)	.	2_665	no
H8A	C1	3.04(4)	.	.	no
H8B	H1A	2.38(7)	.	.	no
H9A	H11A	2.413(17)	.	.	no
H9A	H12A	2.45(2)	.	.	no
H9A	H18	2.592(11)	.	2_665	no
H9B	O1	2.869(6)	.	.	no
H9B	H12B	2.32(3)	.	.	no
H9B	H13	2.473(9)	.	.	no
H9B	O1	2.675(7)	.	2_555	no
H11A	H9A	2.413(17)	.	.	no
H11A	H12A	2.50(4)	.	.	no
H11A	C1	3.10(3)	.	2_665	no
H11A	H1	2.50(3)	.	2_665	no
H11B	C14	2.74(2)	.	.	no
H11B	C19	2.82(3)	.	.	no
H11B	H12C	2.54(4)	.	.	no
H11C	O2	2.715(19)	.	.	no
H11C	O3	2.668(18)	.	.	no
H11C	H2	2.58(4)	.	2_665	no
H12A	H9A	2.45(2)	.	.	no
H12A	H11A	2.50(4)	.	.	no
H12B	H9B	2.32(3)	.	.	no
H12B	H13	2.42(2)	.	.	no
H12C	C4	3.06(3)	.	1_556	no
H12C	C14	2.79(3)	.	.	no
H12C	C15	2.76(5)	.	.	no
H12C	H11B	2.54(4)	.	.	no
H12C	H15	2.59(5)	.	.	no
H13	H9B	2.473(9)	.	.	no
H13	H12B	2.42(2)	.	.	no
H13	H15	2.414(8)	.	.	no
H13	N1	2.877(7)	.	2_555	no
H15	H12C	2.59(5)	.	.	no
H15	H13	2.414(8)	.	.	no
H16	C17	3.089(12)	.	2_565	no
H18	H9A	2.592(9)	.	3_564	no
H19	O3	2.477(7)	.	.	no
H19	C11	3.088(10)	.	.	no

```
loop_
_geom_hbond_atom_site_label_D
_geom_hbond_atom_site_label_H
_geom_hbond_atom_site_label_A
_geom_hbond_distance_DH
_geom_hbond_distance_HA
_geom_hbond_distance_DA
_geom_hbond_angle_DHA
```

```
_geom_hbond_site_symmetry_A
_geom_hbond_publ_flag
#
#D   H   A   D - H  H...A   D...A    D - H...A  symm(A)
#
N1      H1A     O1         0.93(7)    1.90(7)   2.817(5)     171(6)   3_554 yes


# CIF-file generated for  S2106B

#===============================================================================

data_S2106B

#===============================================================================

# 5. CHEMICAL DATA

_chemical_name_systematic
;
 ?
;
_chemical_name_common                 ?
_chemical_melting_point               ?
_chemical_formula_moiety             'C11 H13 Cl2 O3 P'
_chemical_formula_structural          ?
_chemical_formula_sum                'C11 H13 Cl2 O3 P'
_chemical_formula_weight                 295.08
_chemical_compound_source            'see text'

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_source
 P   P    0.1023    0.0942
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 O   O    0.0106    0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Cl  Cl    0.1484    0.1585
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 H   H    0.0000    0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 C   C    0.0033    0.0016
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

#===============================================================================

# 6. CRYSTAL DATA

_symmetry_cell_setting               Orthorhombic
_symmetry_space_group_name_Hall      'P 2ac 2ab'
_symmetry_space_group_name_H-M       'P 21 21 21'

loop_
_symmetry_equiv_pos_as_xyz
 x,y,z
 1/2-x,-y,1/2+z
 1/2+x,1/2-y,-z
 -x,1/2+y,1/2-z

_cell_length_a                         6.4383(10)
_cell_length_b                          10.498(2)
_cell_length_c                          19.571(3)
_cell_angle_alpha                              90
_cell_angle_beta                               90
_cell_angle_gamma                              90
_cell_volume                            1322.8(4)
```

```
_cell_formula_units_Z                    4
_cell_measurement_temperature          150
_cell_measurement_reflns_used          ?
_cell_measurement_theta_min            ?
_cell_measurement_theta_max            ?
_cell_special_details
; ?
;

_exptl_crystal_description             ?
_exptl_crystal_colour                  colourless
_exptl_crystal_size_max               ?
_exptl_crystal_size_mid               ?
_exptl_crystal_size_min               ?
_exptl_crystal_size_rad                ?
_exptl_crystal_density_meas            ?
_exptl_crystal_density_diffrn            1.482
_exptl_crystal_density_method         'Not Measured'
_exptl_crystal_F_000                       608
_exptl_absorpt_coefficient_mu             0.60
_exptl_crystal_density_meas_temp       ?
_exptl_absorpt_correction_type         ?
_exptl_absorpt_process_details         ?
_exptl_absorpt_correction_T_min       ?
_exptl_absorpt_correction_T_max       ?

#===============================================================================

# 7. EXPERIMENTAL DATA

_exptl_special_details
; ?
;
_diffrn_ambient_temperature            150
_diffrn_radiation_wavelength             0.71073
_diffrn_radiation_type                 'Mo K\a'
_diffrn_radiation_source               ?
_diffrn_radiation_monochromator        ?

_diffrn_measurement_device_type        ?
_diffrn_measurement_method             ?
_diffrn_detector_area_resol_mean       ?

_diffrn_standards_number               ?
_diffrn_standards_interval_count       ?
_diffrn_standards_interval_time        ?
_diffrn_standards_decay_%              ?

loop_
_diffrn_standard_refln_index_h
_diffrn_standard_refln_index_k
_diffrn_standard_refln_index_l
 ? ? ?

# number of measured reflections (redundant set)
_diffrn_reflns_number                  35198
_diffrn_reflns_av_R_equivalents         0.0769
_diffrn_reflns_av_sigmaI/netI           0.0256
_diffrn_reflns_limit_h_min               -8
_diffrn_reflns_limit_h_max                8
_diffrn_reflns_limit_k_min              -13
_diffrn_reflns_limit_k_max               13
_diffrn_reflns_limit_l_min              -25
_diffrn_reflns_limit_l_max               25
_diffrn_reflns_theta_min                2.08
_diffrn_reflns_theta_max               27.54
_diffrn_reflns_theta_full              ?
_diffrn_measured_fraction_theta_max    ?
_diffrn_measured_fraction_theta_full   ?
_diffrn_reflns_reduction_process
```

```
;
 ?
;

# number of unique reflections
_reflns_number_total                    3040
# number of observed reflections (> n sig(I))
_reflns_number_gt                       2941
_reflns_threshold_expression         >2sigma(i)

_computing_data_collection           'Locally modified CAD4-Version 5 Software'
_computing_cell_refinement           'SET4 (de Boer & Duisenberg, 1984)'
_computing_data_reduction            'HELENA (Spek, 1997)'
_computing_structure_solution        ?
_computing_structure_refinement      'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics        ?
_computing_publication_material      'PLATON (Spek, 1990)'

#===============================================================================

# 8. REFINEMENT DATA

_refine_special_details
;
 Refinement on F^2 for ALL reflections except those
 flagged by the user for potential systematic errors.
 Weighted R-factors wR and all goodnesses of fit S
 are based on F^2, conventional R-factors R are based
 on F, with F set to zero for negative F^2. The
 observed criterion of F^2 > 2sigma(F^2) is used only
 for calculating -R-factor-obs etc. and is not
 relevant to the choice of reflections for refinement.
 R-factors based on F^2 are statistically about twice
 as large as those based on F, and R-factors based on
 ALL data will be even larger.
;
_refine_ls_structure_factor_coef     Fsqd
_refine_ls_matrix_type               full
_refine_ls_weighting_scheme          'calc       '
_refine_ls_weighting_details
'CALC W=1/[^2^(FO^2^)+(0.0323P)^2^+0.2106P] WHERE P=(FO^2^+2FC^2^)/3'
_refine_ls_hydrogen_treatment        'H-atom refinement: see text '
_refine_ls_extinction_method         none
_refine_ls_abs_structure_details
 'Flack H.D. (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack             -0.01(5)
_refine_ls_number_reflns                 3040
_refine_ls_number_parameters              206
_refine_ls_number_restraints                0
_refine_ls_number_constraints        ?
_refine_ls_R_factor_all                 0.0241
_refine_ls_R_factor_gt                  0.0228
_refine_ls_wR_factor_ref                0.0590
_refine_ls_wR_factor_gt                 0.0582
_refine_ls_goodness_of_fit_ref           1.059
_refine_ls_restrained_S_all              1.059
_refine_ls_shift/su_max                  0.001
_refine_ls_shift/su_mean                 0.000
_refine_diff_density_max                 0.181
_refine_diff_density_min                -0.320
_refine_diff_density_rms                 0.051

#===============================================================================

# 9. ATOMIC COORDINATES AND THERMAL PARAMETERS

loop_
_atom_site_label
_atom_site_type_symbol
_atom_site_thermal_displace_type
```

```
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_U_iso_or_equiv
 Cl7    Cl Uani    0.05830(6)   1.17884(3)   0.98672(2)     1.000   0.0296(1)
 Cl15   Cl Uani    0.49021(7)   1.09130(4)   0.77252(2)     1.000   0.0414(1)
 P1     P  Uani   -0.10188(5)   1.02163(3)   0.96298(2)     1.000   0.0204(1)
 O2     O  Uani  -0.06812(15)   1.00339(9)   0.88419(5)     1.000   0.0219(3)
 O6     O  Uani   0.01990(16)   0.91077(9)   0.99864(4)     1.000   0.0237(3)
 O8     O  Uani  -0.31824(16)  1.02975(11)   0.98338(6)     1.000   0.0301(3)
 C3     C  Uani     0.1407(2)  0.97077(14)   0.85862(7)     1.000   0.0194(3)
 C4     C  Uani     0.2181(2)  0.84918(13)   0.89472(6)     1.000   0.0197(4)
 C5     C  Uani     0.2257(2)  0.87695(14)   0.97171(7)     1.000   0.0230(4)
 C9     C  Uani     0.1243(2)  0.96049(14)   0.78178(7)     1.000   0.0239(4)
 C10    C  Uani    -0.0439(3)  0.89939(15)   0.75146(7)     1.000   0.0297(4)
 C11    C  Uani    -0.0539(3)  0.88149(17)   0.68108(8)     1.000   0.0390(5)
 C12    C  Uani     0.1069(4)  0.92444(18)   0.64040(8)     1.000   0.0424(6)
 C13    C  Uani     0.2735(3)  0.98692(18)   0.66858(8)     1.000   0.0402(5)
 C14    C  Uani     0.2802(3)  1.00619(16)   0.73929(7)     1.000   0.0293(4)
 C16    C  Uani     0.0824(3)  0.73241(14)   0.88070(8)     1.000   0.0269(4)
 C17    C  Uani     0.4426(2)  0.82142(15)   0.87307(7)     1.000   0.0255(4)
 H3     H  Uiso      0.225(2)   1.0376(15)    0.8707(7)     1.000    0.014(4)
 H5A    H  Uiso      0.321(3)   0.9480(17)    0.9811(9)     1.000    0.023(4)
 H5B    H  Uiso      0.263(3)   0.8029(18)    0.9961(8)     1.000    0.034(5)
 H10    H  Uiso     -0.163(3)   0.8764(19)   0.7829(10)     1.000    0.041(5)
 H11    H  Uiso     -0.175(3)   0.8428(19)   0.6637(10)     1.000    0.036(5)
 H12    H  Uiso      0.108(3)     0.911(2)   0.5955(11)     1.000    0.047(6)
 H13    H  Uiso      0.394(4)     1.016(2)   0.6454(11)     1.000    0.054(6)
 H16A   H  Uiso      0.096(3)   0.7091(18)   0.8312(10)     1.000    0.036(5)
 H16B   H  Uiso     -0.076(3)   0.7568(18)    0.8909(8)     1.000    0.031(4)
 H16C   H  Uiso      0.126(3)   0.6637(19)    0.9089(9)     1.000    0.037(5)
 H17A   H  Uiso      0.450(3)   0.8025(18)    0.8238(9)     1.000    0.036(5)
 H17B   H  Uiso      0.536(3)   0.8931(16)    0.8843(8)     1.000    0.024(4)
 H17C   H  Uiso      0.495(3)   0.7465(19)    0.8965(9)     1.000    0.034(5)

loop_
_atom_site_aniso_label
_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
  Cl7      0.0296(2)  0.0238(2)  0.0354(2)  -0.0052(1)  -0.0027(1)  -0.0047(1)
  Cl15     0.0358(2)  0.0459(3)  0.0425(2)   0.0147(2)   0.0091(2)  -0.0092(2)
  P1       0.0189(2)  0.0224(2)  0.0200(2)  -0.0029(1)   0.0024(1)  -0.0016(1)
  O2       0.0176(4)  0.0280(5)  0.0200(4)  -0.0008(4)   0.0009(3)   0.0032(4)
  O6       0.0274(5)  0.0247(5)  0.0189(4)   0.0019(4)   0.0047(4)   0.0005(4)
  O8       0.0201(5)  0.0382(6)  0.0320(5)  -0.0103(5)   0.0068(4)  -0.0024(5)
  C3       0.0167(6)  0.0228(6)  0.0188(6)   0.0004(5)   0.0025(5)   0.0009(5)
  C4       0.0204(7)  0.0204(6)  0.0182(6)   0.0004(5)   0.0002(5)   0.0005(5)
  C5       0.0244(7)  0.0245(7)  0.0202(6)   0.0018(5)  -0.0007(5)   0.0030(6)
  C9       0.0266(7)  0.0266(7)  0.0185(6)   0.0038(5)   0.0014(5)   0.0071(6)
  C10      0.0310(8)  0.0364(8)  0.0218(6)  -0.0017(6)  -0.0018(6)   0.0039(7)
  C11     0.0513(11)  0.0409(9)  0.0249(7)  -0.0046(7)  -0.0094(7)   0.0070(8)
  C12     0.0718(13)  0.0387(9)  0.0167(7)   0.0003(6)   0.0019(8)   0.0141(9)
  C13     0.0584(11)  0.0367(9)  0.0255(8)   0.0093(7)   0.0163(8)   0.0114(8)
  C14      0.0336(8)  0.0288(7)  0.0256(7)   0.0077(6)   0.0063(6)   0.0052(6)
  C16      0.0333(8)  0.0219(6)  0.0255(7)   0.0000(5)   0.0008(6)  -0.0039(6)
  C17      0.0225(7)  0.0287(7)  0.0254(7)   0.0006(6)   0.0005(5)   0.0068(6)

#=============================================================================

# 10. MOLECULAR GEOMETRY

_geom_special_details
;
 Bond distances, angles etc. have been calculated using the
 rounded fractional coordinates. All esds are estimated
```

```
 from the variances of the (full) variance-covariance matrix.
 The cell esds are taken into account in the estimation of
 distances, angles and torsion angles
;

loop_
_geom_bond_atom_site_label_1
_geom_bond_atom_site_label_2
_geom_bond_distance
_geom_bond_site_symmetry_1
_geom_bond_site_symmetry_2
_geom_bond_publ_flag
 Cl7     P1        2.0008(6)                    .       .                yes
 Cl15    C14      1.7463(19)                    .       .                yes
 P1      O2       1.5689(11)                    .       .                yes
 P1      O6       1.5673(10)                    .       .                yes
 P1      O8       1.4516(11)                    .       .                yes
 O2      C3       1.4749(16)                    .       .                yes
 O6      C5       1.4695(17)                    .       .                yes
 C3      C4         1.542(2)                    .       .                no
 C3      C9         1.511(2)                    .       .                no
 C4      C5       1.5355(18)                    .       .                no
 C4      C16        1.530(2)                    .       .                no
 C4      C17      1.5342(19)                    .       .                no
 C9      C10        1.391(2)                    .       .                no
 C9      C14        1.389(2)                    .       .                no
 C10     C11        1.392(2)                    .       .                no
 C11     C12        1.382(3)                    .       .                no
 C12     C13        1.373(3)                    .       .                no
 C13     C14        1.399(2)                    .       .                no
 C3      H3        0.918(15)                    .       .                no
 C5      H5A       0.983(18)                    .       .                no
 C5      H5B       0.943(18)                    .       .                no
 C10     H10         1.01(2)                    .       .                no
 C11     H11         0.94(2)                    .       .                no
 C12     H12         0.89(2)                    .       .                no
 C13     H13         0.95(2)                    .       .                no
 C16     H16A      1.003(19)                    .       .                no
 C16     H16B      1.070(19)                    .       .                no
 C16     H16C      0.951(19)                    .       .                no
 C17     H17A      0.986(18)                    .       .                no
 C17     H17B      0.988(18)                    .       .                no
 C17     H17C      0.971(19)                    .       .                no

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 Cl7     P1      O2        104.92(4)              .       .       .          yes
 Cl7     P1      O6        104.55(4)              .       .       .          yes
 Cl7     P1      O8        112.48(5)              .       .       .          yes
 O2      P1      O6        106.13(5)              .       .       .          yes
 O2      P1      O8        114.23(6)              .       .       .          yes
 O6      P1      O8        113.65(6)              .       .       .          yes
 P1      O2      C3        119.22(8)              .       .       .          yes
 P1      O6      C5        118.08(8)              .       .       .          yes
 O2      C3      C4       109.35(10)              .       .       .          yes
 O2      C3      C9       106.89(10)              .       .       .          yes
 C4      C3      C9       114.80(12)              .       .       .          no
 C3      C4      C5       107.63(11)              .       .       .          no
 C3      C4      C16      113.36(11)              .       .       .          no
 C3      C4      C17      109.59(11)              .       .       .          no
 C5      C4      C16      110.26(11)              .       .       .          no
 C5      C4      C17      106.08(10)              .       .       .          no
 C16     C4      C17      109.65(12)              .       .       .          no
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 O6      C5      C4       111.66(10)             .       .       .          yes
 C3      C9      C10      120.77(12)             .       .       .          no
 C3      C9      C14      121.37(12)             .       .       .          no
 C10     C9      C14      117.80(13)             .       .       .          no
 C9      C10     C11      121.35(16)             .       .       .          no
 C10     C11     C12      119.45(17)             .       .       .          no
 C11     C12     C13      120.65(15)             .       .       .          no
 C12     C13     C14      119.37(17)             .       .       .          no
 Cl15    C14     C9       120.88(11)             .       .       .          yes
 Cl15    C14     C13      117.79(14)             .       .       .          yes
 C9      C14     C13      121.33(16)             .       .       .          no
 O2      C3      H3         105.9(9)             .       .       .          no
 C4      C3      H3         108.9(9)             .       .       .          no
 C9      C3      H3         110.6(9)             .       .       .          no
 O6      C5      H5A       108.2(11)             .       .       .          no
 O6      C5      H5B       104.3(11)             .       .       .          no
 C4      C5      H5A       110.3(10)             .       .       .          no
 C4      C5      H5B       110.4(10)             .       .       .          no
 H5A     C5      H5B       111.8(16)             .       .       .          no
 C9      C10     H10       116.1(11)             .       .       .          no
 C11     C10     H10       122.3(11)             .       .       .          no
 C10     C11     H11       117.0(12)             .       .       .          no
 C12     C11     H11       123.5(12)             .       .       .          no
 C11     C12     H12       121.5(13)             .       .       .          no
 C13     C12     H12       117.8(13)             .       .       .          no
 C12     C13     H13       126.9(13)             .       .       .          no
 C14     C13     H13       113.6(14)             .       .       .          no
 C4      C16     H16A      108.6(11)             .       .       .          no
 C4      C16     H16B      108.6(10)             .       .       .          no
 C4      C16     H16C      109.6(12)             .       .       .          no
 H16A    C16     H16B      108.8(14)             .       .       .          no
 H16A    C16     H16C      110.5(16)             .       .       .          no
 H16B    C16     H16C      110.8(15)             .       .       .          no
 C4      C17     H17A      110.7(11)             .       .       .          no
 C4      C17     H17B      111.5(11)             .       .       .          no
 C4      C17     H17C      110.5(11)             .       .       .          no
 H17A    C17     H17B      110.0(14)             .       .       .          no
 H17A    C17     H17C      106.4(15)             .       .       .          no
 H17B    C17     H17C      107.5(15)             .       .       .          no

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 Cl7     P1      O2      C3         67.32(9)     .       .       .       .   no
 O6      P1      O2      C3       -43.03(10)     .       .       .       .   no
 O8      P1      O2      C3      -169.05(10)     .       .       .       .   no
 Cl7     P1      O6      C5        -69.40(9)     .       .       .       .   no
 O2      P1      O6      C5         41.21(10)    .       .       .       .   no
 O8      P1      O6      C5         167.58(9)    .       .       .       .   no
 P1      O2      C3      C4         55.51(13)    .       .       .       .   no
 P1      O2      C3      C9       -179.66(9)     .       .       .       .   no
 P1      O6      C5      C4        -53.68(13)    .       .       .       .   no
 O2      C3      C4      C5        -60.09(13)    .       .       .       .   no
 O2      C3      C4      C16        62.12(14)    .       .       .       .   no
 C9      C3      C4      C17        64.87(14)    .       .       .       .   no
 O2      C3      C9      C10       -42.48(18)    .       .       .       .   no
 O2      C3      C9      C14       140.51(14)    .       .       .       .   no
 C4      C3      C9      C10        78.97(17)    .       .       .       .   no
 O2      C3      C4      C17      -175.03(10)    .       .       .       .   no
 C9      C3      C4      C5        179.81(14)    .       .       .       .   no
 C9      C3      C4      C16       -57.98(15)    .       .       .       .   no
 C4      C3      C9      C14       -98.04(17)    .       .       .       .   no
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 C17     C4      C5      O6       177.54(11)     .       .       .       .   no
 C16     C4      C5      O6       -63.81(15)     .       .       .       .   no
 C3      C4      C5      O6        60.30(14)     .       .       .       .   no
 C3      C9      C10     C11     -175.50(15)     .       .       .       .   no
 C14     C9      C10     C11          1.6(2)     .       .       .       .   no
 C3      C9      C14     Cl15        -6.3(2)     .       .       .       .   no
 C3      C9      C14     C13      174.42(15)     .       .       .       .   no
 C10     C9      C14     Cl15     176.65(12)     .       .       .       .   no
 C10     C9      C14     C13         -2.7(2)     .       .       .       .   no
 C9      C10     C11     C12          0.4(3)     .       .       .       .   no
 C10     C11     C12     C13         -1.5(3)     .       .       .       .   no
 C11     C12     C13     C14          0.4(3)     .       .       .       .   no
 C12     C13     C14     Cl15    -177.65(15)     .       .       .       .   no
 C12     C13     C14     C9           1.7(3)     .       .       .       .   no

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 Cl7     Cl7      3.5868(9)                     .     3_577                 no
 Cl7     O8      3.2145(13)                     .     3_577                 no
 Cl7     Cl7      3.5868(9)                     .     3_477                 no
 Cl15    C17     3.4632(17)                     .       .                   no
 Cl15    C10       3.637(2)                     .     1_655                 no
 Cl7     H5A      2.957(18)                     .       .                   no
 Cl7     H12        3.11(2)                     .     4_556                 no
 Cl7     H3       2.917(14)                     .       .                   no
 Cl15    H3       2.632(13)                     .       .                   no
 Cl15    H17B     3.033(16)                     .       .                   no
 Cl15    H17A     2.936(18)                     .     4_656                 no
 P1      C16     3.6358(17)                     .       .                   no
 P1      H16B     3.122(18)                     .       .                   no
 P1      H17B     3.103(18)                     .     1_455                 no
 O8      C5      3.3536(18)                     .     1_455                 no
 O8      Cl7     3.2145(13)                     .     3_477                 no
 O2      H16B     2.593(19)                     .       .                   no
 O2      H10        2.47(2)                     .       .                   no
 O2      H17B     2.799(19)                     .     1_455                 no
 O6      H5B      2.789(19)                     .     3_467                 no
 O6      H17C     2.639(19)                     .     3_467                 no
 O6      H16B     2.728(17)                     .       .                   no
 O8      H5A      2.477(19)                     .     1_455                 no
 O8      H17B     2.588(17)                     .     1_455                 no
 C5      O8      3.3536(18)                     .     1_655                 no
 C10     C16       3.183(2)                     .       .                   no
 C10     Cl15      3.637(2)                     .     1_455                 no
 C12     C16       3.480(3)                     .     4_556                 no
 C13     C16       3.581(3)                     .     4_556                 no
 C14     C17       3.422(2)                     .       .                   no
 C16     C10       3.183(2)                     .       .                   no
 C16     C12       3.480(3)                     .     4_546                 no
 C16     C13       3.581(3)                     .     4_546                 no
 C16     P1      3.6358(17)                     .       .                   no
 C17     C14       3.422(2)                     .       .                   no
 C17     Cl15    3.4632(17)                     .       .                   no
 C9      H17A     2.797(19)                     .       .                   no
 C9      H16A     2.817(19)                     .       .                   no
 C10     H16A     2.690(19)                     .       .                   no
 C12     H16C       3.08(2)                     .     4_556                 no
 C14     H17A     2.916(19)                     .       .                   no
 C16     H10        2.91(2)                     .       .                   no
 H3      Cl7      2.917(14)                     .       .                   no
 H3      Cl15     2.632(13)                     .       .                   no
 H3      H5A        2.44(2)                     .       .                   no
 H3      H17B       2.53(2)                     .       .                   no
 H5A     Cl7      2.957(18)                     .       .                   no
 H5A     O8       2.477(19)                     .     1_655                 no
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 H5A     H3          2.44(2)                    .       .                  no
 H5A     H17B        2.42(2)                    .       .                  no
 H5B     H16C        2.41(3)                    .       .                  no
 H5B     H17C        2.53(3)                    .       .                  no
 H5B     O6        2.789(19)                    .     3_567                no
 H5B     H16B        2.52(2)                    .     3_567                no
 H10     O2          2.47(2)                    .       .                  no
 H10     C16         2.91(2)                    .       .                  no
 H10     H16A        2.60(3)                    .       .                  no
 H10     H16B        2.52(3)                    .       .                  no
 H12     Cl7         3.11(2)                    .     4_546                no
 H16A    C9        2.817(19)                    .       .                  no
 H16A    C10       2.690(19)                    .       .                  no
 H16A    H10         2.60(3)                    .       .                  no
 H16A    H17A        2.49(3)                    .       .                  no
 H16B    P1        3.122(18)                    .       .                  no
 H16B    O2        2.593(19)                    .       .                  no
 H16B    O6        2.728(17)                    .       .                  no
 H16B    H10         2.52(3)                    .       .                  no
 H16B    H5B         2.52(2)                    .     3_467                no
 H16C    H5B         2.41(3)                    .       .                  no
 H16C    H17C        2.54(3)                    .       .                  no
 H16C    C12         3.08(2)                    .     4_546                no
 H17A    C9        2.797(19)                    .       .                  no
 H17A    C14       2.916(19)                    .       .                  no
 H17A    H16A        2.49(3)                    .       .                  no
 H17A    Cl15      2.936(18)                    .     4_646                no
 H17B    Cl15      3.033(16)                    .       .                  no
 H17B    P1        3.103(18)                    .     1_655                no
 H17B    O2        2.799(19)                    .     1_655                no
 H17B    O8        2.588(17)                    .     1_655                no
 H17B    H3          2.53(2)                    .       .                  no
 H17B    H5A         2.42(2)                    .       .                  no
 H17C    H5B         2.53(3)                    .       .                  no
 H17C    H16C        2.54(3)                    .       .                  no
 H17C    O6        2.639(19)                    .     3_567                no

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#
#D   H   A   D - H  H...A   D...A    D - H...A  symm(A)
#
C3      H3      Cl15     0.918(15)  2.632(13) 3.0829(15)  111.1(10)     .   yes
C5      H5A     O8       0.983(18)  2.477(19) 3.3536(18)  148.4(15)   1_655 yes
C16     H16B    O2       1.070(19)  2.593(19) 3.0061(19)  102.2(12)     .   yes
C17     H17B    O8       0.988(18)  2.588(17) 3.4373(19)  144.0(12)   1_655 yes
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